

Air Resources Board

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TO:

John Sanders, Ph.D., Chief

Environmental Monitoring Branch
Department of Pesticide Regulation

FROM:

Jeff Cook, Chief

Quality Management Branch

Monitoring and Laboratory Division

DATE:

June 8, 2004

SUBJECT:

FINAL REPORT FOR THE 2002 APPLICATION AIR MONITORING FOR

METAM SODIUM BREAKDOWN PRODUCTS IN VENTURA COUNTY

Attached is the final "Report for Air Monitoring Around a Tarped, Drip Irrigation Application of Metam Sodium in Ventura County – Spring 2002." We received your May 6, 2004 comments on the draft report and have made the recommended changes and corrections. The report and separate volume of appendices for the report have also been forwarded to Randy Segawa and Pam Wofford of your staff.

If you or your staff have questions or need further information, please contact me at (916) 322-3726 or via e-mail at icook@arb.ca.gov or Kevin Mongar at (916) 322-2449 or via e-mail at kmongar@arb.ca.gov.

Attachment/Separate Appendices

cc: See next page

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California Environmental Protection Agency Air Resources Board

State of California
California Environmental Protection Agency
AIR RESOURCES BOARD

Report for Air Monitoring
Around a Tarped, Drip Irrigation Application
of Metam Sodium in Ventura County
Spring 2002

Prepared by
Operations Planning and Assessment Section
Quality Management Branch
Monitoring and Laboratory Division

Project No. P-02-001

June 1, 2004

This report has been reviewed by the staff of the California Air Resources Board and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Air Resources Board, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

Monitoring Report Approval

Title:

Report for Air Monitoring Around a Tarped, Drip Irrigation

Application of Metam Sodium in Ventura County - Spring 2002

Prepared by:

Kevin Mongar, Air Pollution Specialist

Approval:

The following monitoring report has been reviewed and approved

by the Monitoring and Laboratory Division.

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10 - ⊤ . [Date

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Executive Summary

Report for the Air Monitoring Around a Tarped, Drip Irrigation Application of Metam Sodium in Ventura County- Spring 2002

This report presents the results of air monitoring for breakdown products of metam sodium around a drip irrigation application. The monitoring was conducted in Ventura County, from May 7 to 12, 2002, around a pre-plant application of metam sodium on an approximately 10 acre bedded/tarped field prior to planting bell peppers. Monitoring was conducted for metam sodium as the breakdown products methyl isothiocyanate (MITC), methyl isocyanate (MIC), hydrogen sulfide (H₂S), and carbon disulfide (CS₂).

The study included a total of seven sampling periods (one background period and six sampling periods) at eight sampling sites. Samples were collected using charcoal adsorbent tubes for MITC and XAD-7 adsorbent tubes for MIC at all eight sampling sites during each sampling period. Samples were collected using Silcosteel® canisters for carbon disulfide at four sampling sites (east (E), north (N), west (W) and south (S) sites) during each of the sampling periods. On-site monitoring was conducted for hydrogen sulfide using a Jerome portable H₂S monitor at each sampling site during each sample change-out.

MIC

All results for MIC were less than the method detection limit (MDL) of 0.42 micrograms per cubic meter of sampled air (ug/m³). Five samples were invalidated due to sampling problems.

Carbon Disulfide

All results for carbon disulfide were "detected" (Det) except for three samples collected during the second sampling period which were <MDL. The term "detected" means that the result was greater than the MDL but less than the estimated quantititation limit (EQL). The results of three background samples were also "Det" for CS_2 (one sample was invalidated). However, since the laboratory system blanks also showed detectable amounts of CS_2 only results above the EQL should be considered valid and reportable. No results were above the EQL, which ranged from 2.7 to 7.3 ug/m³ depending on the dilution per canister.

Hydrogen Sulfide

All results for hydrogen sulfide were less than the EQL of 15 ppbv.

MITC

Concentrations of MITC ranged from <MDL to 35 ug/m³ (12 parts per billion by volume (ppbv)). The highest concentration was observed at the west sampling site during the third sampling period (overnight after application day).

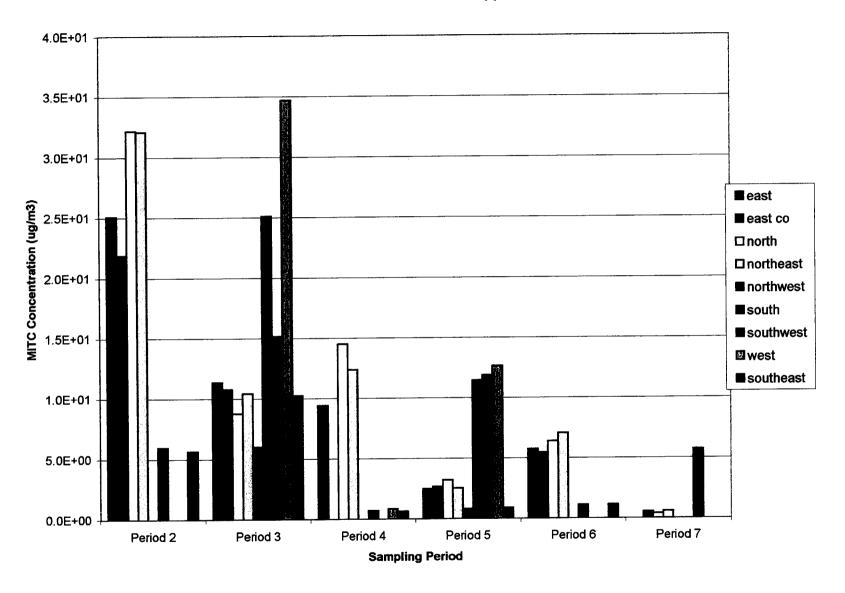
Four samples were collected for the background period (i.e., prior to application) from

the east, north, west and south sites. The MITC results from three background samples were <MDL and the result from the north site was not valid due to a sampling problem.

Of the 48 application samples collected for MITC, excluding spikes, blanks, background samples and the lower of each pair of collocated samples, 35 sample results were above the EQL, no sample results were "detected", eight sample results were <MDL, and five samples were invalidated due to sampling problems.

The bar graph attached to this Executive Summary illustrates the MITC results of the metam sodium tarped, drip application study.

MITC Results- Metam Sodium Application



Acknowledgments

Assistance was provided by staff of the Ventura County Agricultural Commissioner's Office. Staff of the Source Test Section collected the samples. David Frisk coordinated the field work. Jim Omand, Michael Orbanosky and T.E. Houston, Ph.D., of the ARB Special Analysis Section laboratory performed the method development and chemical analyses. Lynn Baker of the ARB Stationary Source Division provided comments on the monitoring protocol and report.

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Report for Air Monitoring Around a Tarped, Drip Irrigation Application of Metam Sodium Spring 2002

I. <u>Introduction</u>

At the request of the California Department of Pesticide Regulation (DPR) (June 28, 2000, Memorandum, Helliker to Lloyd, and July 25, 2001, Memorandum, Sanders to Cook), the Air Resources Board (ARB) staff determined airborne concentrations of the breakdown products of the pesticide metam sodium around a tarped drip application. This monitoring was done to fulfill the requirements of Assembly Bill 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5) which requires the ARB "to document the level of airborne emissions...of pesticides which may be determined to pose a present or potential hazard..." when requested by the DPR. The monitoring was conducted in Ventura County, from May 7 to 12, 2002, around an application of metam sodium on an approximately 10 acre field (pre-plant for bell peppers). Monitoring was conducted for metam sodium as the breakdown products methyl isothiocyanate, methyl isocyanate, hydrogen sulfide and carbon disulfide.

The sampling and analysis followed the procedures outlined in 1) the monitoring protocol (Appendix 1 of the separate volume of Appendices), 2) the quality assurance guidelines described in the "Quality Assurance Plan for Pesticide Air Monitoring" (May 11, 1999 version), 3) the "Standard Operating Procedure, Sampling and Analysis Methyl Isothiocyanate (MITC) in Application Air Monitoring using Gas Chromatography/Mass Selective Detector" (page 63 of the Appendices), 4) the "Standard Operating Procedure, Sampling, and Analysis of Methyl Isocyanate in Application Air using High Performance Liquid Chromatography with a Fluorescence Detector," (page 70 of Appendices), and 5) the "Standard Operating Procedure, Sampling, and Analysis of Carbon Disulfide in Silco™ Canisters Using a Varian Stand Alone Cryogenic Sampler" (page 86 of Appendices).

II. Sampling

Monitoring for methyl isocyanate (MIC) and methyl isothiocyanate (MITC) was conducted with sampling tubes. Monitoring for carbon disulfide was conducted using Silcosteel® canisters. Monitoring for hydrogen sulfide was conducted using a portable sampler (Jerome sampler).

MIC and MITC Sampling:

The sampling methods for two of the compounds require passing measured quantities of ambient air through adsorbent sampling tubes. For MIC, the tubes were 8 mm x 110 mm, XAD-7, 1-(2-pyridyl)piperazine coated, with 400 mg in the primary section and 200

mg in the secondary section (Supelco special order). For MITC, the tubes were 8 mm x 110 mm, coconut shell charcoal with 400 mg in the primary section and 200 mg in the secondary section (SKC catalogue #226-09).

Sample collection for MIC was conducted using a flow rate of 75 standard cubic centimeters per minute (sccpm). For MITC a flow rate of 2.5 standard liters per minute (slpm) was used. Immediately after sampling, the tubes were capped, labeled, placed in culture tubes and stored and transported in an insulated container with dry ice to the ARB laboratory in Sacramento.

Each sample train consisted of an adsorbent tube, Teflon fittings and tubing, rain/sun shield, needle valve, train support and a 12 volt DC vacuum pump (Figure 1). Tubes were prepared for use by breaking off the sealed glass end and immediately inserting the tube into the Teflon fitting. The tubes were oriented in the sample train according to a small arrow printed on the side of the tube indicating the direction of flow. A needle valve with a range of 0.5-4 slpm was used to control sample flow for the MITC sampling and a needle valve with a range of 25-500 ccpm was used to control the flow for the MIC sampling. The flow rates were set using calibrated digital mass flow meters (MFM) before the start of each sampling period. A MFM scaled from 0-5 slpm was used for MITC and a 0-100 sccpm MFM was used for the MIC samplers. The flow rate was also checked and recorded, using the MFM, at the end of each sampling period. Samplers were leak checked prior to each sampling period with the sampling tubes installed. Any change in flow rates was recorded on the field log sheet. The pesticide sampling procedures for adsorbent tubes are included in Appendix I (page 41 of Appendices).

Caution was used during field monitoring, transportation, storage, and lab analysis to minimize exposure of samples to sunlight in order to prevent photo-degradation of MITC and MIC.

Carbon Disulfide Sampling:

Integrated ambient air samples were collected for carbon disulfide using passive air sampling into evacuated six liter, Silcosteel® canisters purchased from Restek Corporation. The flow rate of 3 sccpm was set and measured using a 0-10 sccpm mass flow meter. The sampling system was operated continuously with the exact operating interval recorded in the log-book and on the field data sheets. The canister vacuum reading was recorded at the start and end of each sampling period using the -30 to 0 inch Hg gauge on the passive sampler. The canister vacuum reading was also measured using a more accurate gauge in the lab before and after transport to/from the field. The laboratory gauge readings were used to calculate the sample volume collected.

The critical orifice flow controllers (Silcosteel treated Veriflo SC423XL) were attached to the valve fitting on each CS2 canister using a Silcosteel® treated swagelock connector (Figure 1, page 12 of the Appendices). A six foot section of 1/8 inch O.D., Silcosteel tubing was attached to the inlet end of an in-line, 5 micron filter, which was attached to

the inlet end of the flow controller. The inlet end of the tubing was bent into a "U" shape to prevent rain or other materials from entering. At the end of each sampling period the canisters were placed in shipping containers with a sample identification/chain of custody sheet and transported as soon as possible to the ARB Monitoring and Laboratory Division laboratory for analysis. The samples were stored at ambient laboratory temperature prior to analysis.

When using a critical orifice flow restrictor for passive, integrated canister sampling, the potential decrease in flow rate as the vacuum in the canister changes must be taken into account. The flow control device used for the study (Veriflo SC423XL from Restek Corporation) is designed to regulate and maintain a constant flow as the vacuum in the canister decreases. The manufacturer specifications indicate that the controller is capable of maintaining a continuous low flow with vacuum ranges from -29.9 to -5 inch Hg. The in-line filter helps prevent particles from entering the critical orifice of the flow controller, which could clog the critical orifice and affect the flow through the controller. The manufacturer specifications indicate that the outside temperature can have a slight effect on the flow rate. For example, there could be approximately 6 percent drop in flow when the ambient temperature changes from 80 °F to 125 °F. The ambient temperature ranged from 45 °F to 70 °F during the study.

The pesticide ambient sampling procedures for canisters are included on page 38 of the Appendices. The canister sampling field log sheet and canister data sheet are included on page 44 of the Appendices. These forms were used to record start and stop times, start and stop vacuum readings, sample identifications, weather conditions, field operator's initials and any other significant data.

Hydrogen Sulfide Sampling:

Hydrogen Sulfide was sampled with a portable Jerome 631-X Hydrogen Sulfide Analyzer. A 25 second sample was taken each time the cartridges were changed out and at approximately mid-way through the application. The Jerome Analyzer samples at 150cc/min and for the expected range of 0.10 to 0.99 ppm must sample for 25 seconds. Prior to first use and after each morning and evening recovery the sampler was regenerated to assure the sensor was zeroed prior to use. The principle of operation involves hydrogen sulfide reacting with a thin gold film by increasing the electrical resistance in proportion to the mass of hydrogen sulfide present in the atmosphere.

III. Application Monitoring

The DPR's monitoring recommendation (July 25, 2001 memo, Sanders to Cook, Updated Monitoring Recommendations for 2001) directed that "monitoring for metam sodium be a drip irrigation application at a site using the highest allowed rates of use (i.e., about 318 pounds active ingredient (AI) per treated (raised bed area) acre)". Candidate fields for application monitoring were to be 10 acres or larger. The crop type

for the application study was not specified by the DPR.

The sampling schedule recommended by the DPR consisted of samples collected during daylight and overnight periods as shown below in Table 1.

Table 1 Application Sampling Schedule

Sample period begins Sample duration time 24 hours if possible; minimum 12 hours Background (pre-application) (if <24 hours must meet 24-hour Target EQL) Start of application until 1 hour before During application and post –application sunset Overnight (until 1 hour after sunrise) 1 hour before sunset 1 hour after sunrise Daytime (until 1 hour before sunset) 1 hour before sunset Overnight (until 1 hour after sunrise) 1 hour after sunrise Daytime (until 1 hour before sunset) Overnight (until 1 hour after sunrise) 1 hour before sunset

In the event that application occured at night, the alternate day-night schedule was to be followed. If the fumigation took two or more days, samples were to be collected during the overnight period separating the applications and the overnight/daytime schedule was then to be followed from the last day of application.

A field of approximately 10 acres was chosen for the application monitoring site. Refer to Figure 2 for a diagram of the application site and to Figure 3 for a topographic map of the area. Refer to Appendix IV (page 98 of Appendices) for a copy of the notice of intent to apply restricted materials. Table 2 summarizes the application information.

Table 2 Application Information

Location:

Ventura County, off Somis Road

R/T/S:

20W/2N/19

Field Size:

Approximately 10 acres

(8.84 acres based on measurements)

Treated Area (bed top):

3.9 acres

(30" bed top, 68" beds on center)

Tarp Width per Row:

Approximately 34"

Product Applied:

Sectagon 42

42.2% Active Ingredient (AI) by weight

Type of Application:

drip chemigation, tarped bed Soil, bell pepper pre-plant

Commodity: Application Rate:

239 lbs. Al/acre

(500 gal)(4.22 lbs. Al/gal)/(8.84 acres)

Actual Application Rate:

541 lbs Al/acre

(treated area)

(500 gal)(4.22 lbs. Al/gal)/(3.9 acres)

Grower/Applicator:

Nishimori Farms/Western Farm Service

Referring to Figure 2, for the MIC and MITC adsorbent tube samples, eight samplers were positioned, one on each side of the field and one at each corner. A ninth replicate sampler was collocated at the east sampling position. For the CS₂ canister samples, four samplers were positioned, one on each side (middle) of the field.

The samplers were located at approximately 60 to 65 feet from the edge of the field except at the south side of the field which was positioned 38 feet from the edge. Due to irregular row lengths at the southwest corner, a 90 degree corner was estimated and the sampler positioned 65 feet diagonally from that estimated corner. Refer to Appendix IX for additional notes regarding the site.

Table 3 lists the GPS coordinates of the field corners and sampling locations. Trees (approximately 30 to 40 feet high) were spread out along the south side of Somis road. All samplers were positioned at the same elevation relative to the field except the Northwest sampler which was elevated 5 feet. All sampler inlets were approximately 2 meters above the ground.

Table 3 Metam Sodium Application 5/7/01 through 5/12/01 Field Corners and Sampler Waypoints

Field Corners:

NEC: Northeast Corner = N 34° 15.1620', W 119° 00.1090'

SEC: Southeast Corner = N 34° 15.0600', W 119° 00.1010'

SWC: Southwest Corner = N 34° 15.0030', W 119° 00.1920'

NWC: Northwest Corner = N 34° 15.1580', W 119° 00.2030'

Sampler Positions:

N: Waypoint = N 34° 15.1710′, W 119° 00.1560′

NE: Waypoint = N 34° 15.1710', W 119° 00.1010'

E: Waypoint = N 34° 15.1120', W 119° 00.0920'

SE: Waypoint = N 34° 15.0540', W 119° 00.0910'

S: Waypoint = N 34° 15.0210', W 119° 00.1440'

SW: Waypoint = N 34° 14.9920', W 119° 00.2030'

W: Waypoint = N 34° 15.0760', W 119° 00.2110'

NW: Waypoint = N 34° 15.1570′, W 119° 00.2130′

MET: Waypoint = N 34° 14.9930', W 119° 00.1910'

Background samples were taken at the W, N, E and S positions to establish if any of the metam sodium breakdown products were detectable in the air before the application (i.e., from nearby applications). The background samples were collected from 1000 to 1015, May 7 to 8, 2002 (24.25 hours). The application was conducted from 0720 to 1000 on May 9, 2002. The chemigation procedure was conducted through drip lines, positioned mid-bed under the plastic tarp. The entire field was chemigated at the same time. Table 4 lists the approximate sampling periods.

Table 4
Application Sampling Periods

<u>Period</u>	Approx. # Hours	<u>Date</u>	<u>Time</u>
Background	24.25 hours	5/7-8/02	1000 to 1015
2 (daytime)	12 hours	5/9/002	0700 to 1900
3 (overnight)	11.5 hours	5/9-10/02	1900 to 0630
4 (daytime)	11.5 hours	5/10/002	0630 to 1800
5 (overnight)	12.5 hours	5/10-11/02	1800 to 0630
6 (daytime)	11.5 hours	5/11/002	0630 to 1800
7 (overnight)	12.5 hours	5/11-12/02	1800 to 0630

The meteorological station was oriented toward true north and was positioned 71 feet to the south of the southwest corner of the field. The meteorological station was set up, at a height of 21 feet, to determine wind speed and direction, air temperature, barometric pressure and relative humidity. Appendix V (page 99 of the Appendices) lists the meteorological station data in 15-minute averages for the test period. ARB staff noted the degree of cloud cover on the sample log sheet whenever sample cartridges were changed. The conditions were clear to partly cloudy during the study period.

IV. Analytical Methodology

The sampling and analysis method (SOP) and validation results for MITC are included in the laboratory report (page 65 of the Appendices). The MITC method consists of sampling with charcoal cartridges followed with GC analysis with mass selective detector. The DPR recommended a target 24-hour estimated quantitation limit (EQL) of 0.5 ug/m³ for MITC. The SOP specifies an EQL of 0.17 ug/ml, which corresponds to 0.14 ug/m³ for MITC for a 24-hour sample collected at 2.5 slpm.

The SOP and method validation results for MIC are included in the laboratory report (page 71 of the Appendices). The MIC method consists of sampling with 1-(2-pyridyl)piperazine coated XAD-7 resin cartridges followed by HPLC analysis with fluoresence detector. The DPR recommended a target 24-hour EQL of 0.1 ug/m³ for MIC. The SOP specifies an EQL of 0.015 ug/ml, which corresponds to 0.42 ug/m³ for MIC for a 24-hour sample collected at 75 sccpm.

The SOP and method validation results for CS₂ are included in the laboratory report (page 86 of the Appendices). The procedures are based on USEPA Method TO-15 and consist of cryogenic pre-concentration of an aliquot of a canister whole air sample followed by GC/MS analysis. The canisters arrive from the field at sub-ambient pressure and are pressurized (diluted) in the laboratory before analysis. The DPR recommended a target 24-hour EQL of 15 ug/m³ for carbon disulfide. The SOP specifies an EQL of from 2.7 to 7.3 ug/m³ for CS₂, depending on the exact dilution per canister.

Hydrogen sulfide was sampled with a portable Jerome 631-X Hydrogen Sulfide Analyzer. According to the operating manual, the detection limit of the Jerome Analyzer is 0.003 ppmv and its range is 0.001 to 50 ppmv. According to standard practice for the pesticide air monitoring program, the EQL is assigned as 5 times the detection limit which calculates to 0.015 ppmv (15 ppbv) for hydrogen sulfide. The DPR's requested quantitation limit was 5 ug/m³ (0.004 ppm, or 4 ppbv).

V. Application Monitoring Results

The monitoring study included a total of 7 sampling periods (1 background period and 6 sampling periods). Samples were collected and analyzed for MITC and MIC at all sampling sites during each period. Samples were collected and analyzed for carbon disulfide at the east (E), north (N), west (W) and south (S) sites during each of the sampling periods. On-site monitoring was conducted for hydrogen sulfide at each sampling site during each sample change-out (i.e., sample times correspond approximately to the times listed in Table 4).

MIC

Referring to Table 3 of the MIC laboratory report (page 58 of the Appendices), all results for MIC were <MDL. Since the results were all <MDL they have not been summarized nor presented further in this report. Note that five samples were invalidated due to sampling problems.

Carbon Disulfide

Referring to Table 3 of the CS₂ laboratory report (page 83 of the Appendices), all results for carbon disulfide were "Det" (>MDL but <EQL) except 3 samples collected during period 2 which were <MDL. The results of the 4 background samples were "Det". However, the laboratory report states (page 81 of Appendices) that system blanks also "showed detectable amounts of CS2." Therefore, only results above the EQL should be considered valid and reportable. Since the application results were all <EQL they have not been summarized nor presented further in this report.

Hydrogen Sulfide

Referring to Appendix VII (page 117 of the Appendices), all results for hydrogen sulfide were less than the EQL of 15 ppbv. Since the results were all <EQL they have not been summarized nor presented further in this report.

MITC

Table 5 of this report presents the results of application air monitoring for MITC in units of ug/m³ and parts per billion by volume (ppbv). A summary of the results is presented in Table 6 and as associated with wind roses (wind speed and direction) in Figures 4 through 10. For period 7 (Figure 10) the met data was only collected through 2400 on May 11, 2002. Thus the wind rose does not represent a complete summary of the wind patterns during that period (i.e., data not available for the last 6.5 hours of that period).

The equation used to convert MITC air concentration results from units of ug/m³ to units of pptv at 1 atmosphere and 25 °C is shown below.

ppbv =
$$(ug/m^3) \times (0.0820575 \text{ liter-atm/mole-}^\circ\text{K})(298^\circ\text{K}) = (0.334) \times (ug/m^3)$$

(1 atm)(73 gram/mole)

Four samples were collected for the background period (i.e., prior to application) from the east (E), north (N), west (W) and south (S) sites. The MITC results from three of the background samples were <MDL and one was not valid due to a sampling problem.

Of the 48 application samples collected for MITC, excluding spikes, blanks, background samples and the lower of each pair of collocated samples, 35 sample results were above the EQL, no sample results were "detected", eight sample results were <MDL, and five samples were invalidated due to sampling problems.

The highest concentration, 35 ug/m³ (12 ppbv), was observed at the W (west) sampling site during the third sampling period (overnight after application day).

No sample results have been adjusted or corrected for recoveries of quality assurance spike samples.

VI. Field Quality Control

Field quality assurance for the application monitoring included the following for each of the :

- 1) Four field spikes obtained by sampling ambient air at the application monitoring site. The field spikes were obtained by sampling ambient air during the background monitoring (i.e., collocated with a background sample at the same environmental and experimental conditions).
- 2) Four trip spikes prepared at the same level as the field spikes. The trip spikes were labeled, recorded on the field log-sheet, and transported along with the field spikes and application samples.
- 3) Four lab spikes prepared at the same level as the field and trip spikes.

 The lab spikes remained in the laboratory freezer and were extracted and analyzed along with the field and trip spikes.
- 4) Collocated (replicate) samples taken for all sampling periods (except the background period) at one sampling location (E).
- 5) Trip blanks were supposed to be obtained, labeled, recorded on the field log-sheet, and transported and submitted along with the field spikes and

application samples. No trip blanks were submitted.

- The battery operated mass flow meters used to set and check the sampling flow rate were calibrated by the ARB's Quality Assurance Section (QAS).
- A flow audit of each sampler was performed by the Quality Assurance Section (QAS) on August 30, 2002, at the MLD's 5th Street warehouse facility. All pesticide sampler flow rates were within the QAS's control limit of ±10 percent of "true" flow.
- 8) The Jerome H2S monitor was calibrated prior to the test and the calibration summary is included as Appendix VIII (page 130 of Appendices).

VII. Quality Control Results

A. Trip Blanks

The purpose of collecting trip spikes is to help assess any contamination or interference that may be caused by the sampling media, sample transportation, and the analytical process.

No trip blank samples were submitted. Field staff inadvertently failed to return trip blank samples.

B. Collocated Sample Results

The relative percent difference (RPD) of the collocated results provides an indication of the precision of the monitoring method (i.e., the lower the RPD the better the precision). RPD is calculated as follows: RPD=(| difference |/average) x 100.

Referring to Table 7, four collocated pairs of samples for the application study had both MITC results above the EQL. The RPD of the data pairs ranged from 5 percent to 14 percent, indicating acceptable precision for the MITC sampling and analyses.

C. Laboratory, Trip and Field Spikes

The purpose of collecting spiked samples is to assess the accuracy (percent recovery) of the sampling and analytical methods. The field spikes are collected by sampling ambient air through the previously spiked cartridges at one of the sampling sites during the background sampling. Thus, the field spikes provide an assessment of the accuracy of the entire method and are collected under the same environmental and experimental conditions as those occurring at the time of ambient sampling. The lab

and trip spikes are used to confirm the field spike results or to help identify the source of losses (problems) when they occur in the field spikes.

Laboratory, trip, and field spikes were prepared by spiking a known amount of the target compound onto the appropriate cartridges. The spikes were made and collected in sets of four.

The laboratory spikes were placed immediately in a freezer and kept there until extraction and analysis. The trip and field spikes were kept in the lab freezer until transported to the field. The trip spikes were kept on dry ice in an ice chest (the same one used for samples) during transport to and from the field and at all times while in the field except log-in and labeling. The extraction and analysis of each set of laboratory, trip and field spikes normally occurs at the same time. The collocated (unspiked) background sample result, if above the EQL, was subtracted from the field spike sample result before calculation of percent recovery of the analytes.

The lab, trip and field spike results (average percent recovery) are summarized and discussed below.

	MIC	MITC	CS2 (Low)	CS2 (high)
Lab	100%	59%/80%*	97%	102%
Trip	95%	55%	99%	104%
Field	92%	56%	99%	102%

^{*59} percent using extraction solvent made prior to May 14, 2002; 80 percent using fresh extraction solvent.

MIC

The laboratory report states that the spike results for MIC are likely influenced by interferences. The laboratory report states (page 54 of Appendices):

"The MIC analysis has interferences arising from the derivatizing agent. It is difficult with the current method to separate the MIC derivative baseline to baseline from the interference. The apparent high recoveries of the field, trip and laboratory spikes are a result of this interference."

The inference is that the spike results would be lower without the interference.

- 1) <u>Laboratory Spikes:</u> The MIC laboratory spike results for the application study are listed in Table 11 of Appendix II (page 62 of Appendices). Each of the spike cartridges was spiked with 0.236 ug/ml of MIC. The average recovery for MIC for the application lab spikes was 100 percent.
- 2) <u>Trip Spikes:</u> The trip spike results for the application study are listed in Table 8 of Appendix II (page 61 of Appendices). Each of the cartridges was spiked with 0.236 ug/ml of MIC. The average recovery for MIC for the application trip spikes was 95

percent. These results are consistent with the lab spike results and indicate that the sample transport, storage and analytical procedures used in this study produce acceptable results for MIC.

3) Field Spikes: The field spike results for the application study are listed in Table 7 of Appendix II (page 61 of Appendices). Each of the spike cartridges was spiked with 0.236 ug/ml of MIC. The average recovery for MIC for the application lab spikes was 92 percent. Two of the field spikes (log numbers 3 and 7) had end flow rates below the validation criteria of ±25 percent from 75 sccpm. The field spike results are consistent with the lab and trip spike results and indicate that the sampling, sample transport, storage and analytical procedures used in this study produce acceptable results for MIC.

MITC

The laboratory report states (page 54 of the Appendices):

"No problems occurred during the analysis of the MITC or MIC application samples. The MITC recovery of the field, trip and laboratory spikes in the first analytical sample batch was low at 55-58%. Prior to the application study, using the mixed solvent of 0.1% CS2/EA indicated a recovery for spiked field samples at greater than 80%. After the analysis of the first batch, staff looked at recoveries for several samples extracted with the initial solvent, newly prepared solvent, and the previous extraction solvent, dichloromethane. The results indicated that the extraction solvent needed to be prepared fresh for the sample batches to insure adequate recovery. An additional set of laboratory spikes were run with the balance of the samples with a recovery of 80.5+3.0%."

The batch of samples analyzed on May 14, 2002 were processed with the "initial solvent" (see page 54 of Appendices).

- 1) <u>Laboratory Spikes:</u> The MITC laboratory spike results for the application study are listed in Table 10 of Appendix II (page 62 of Appendices). Each of the spike cartridges was spiked with 7.94 ug/ml of MITC. The average recovery for MITC for the application lab spikes was 59 percent using the extraction solvent prepared prior to 5/14/02 and 80 percent using the more recently prepared solvent.
- 2) <u>Trip Spikes:</u> The trip spike results for the application study are listed in Table 8 of Appendix II (page 63 of Appendices). Each of the cartridges was spiked with of 7.94 ug/ml MITC. The average recovery for MITC for the application trip spikes was 55 percent using the extraction solvent prepared prior to 5/14/02. These results are consistent with the lab spike results and indicate that the sample transport, storage and analytical procedures used in this study produce acceptable results for MITC.
- 3) <u>Field Spikes:</u> The field spike results for the application study are listed in Table 6 of Appendix II (page 63 of Appendices). Each of the cartridges was spiked with 7.94

ug/ml of MITC. The average recovery for MITC for the application field spikes was 56 percent using the extraction solvent prepared prior to 5/14/02. The field spike results are consistent with the lab and trip spike results and indicate that the sampling, sample transport, storage and analytical procedures used in this study produce acceptable results for MITC.

Carbon Disulfide

- 1) <u>Laboratory Spikes:</u> The laboratory spike results for the application study are listed in Table 6 of Appendix II (page 84 of Appendices). The canisters were spiked with 2 levels (2 each) of carbon disulfide, 7.59 ug/m³ (low) and 38.4 ug/m³ (high). The average recovery for carbon disulfide for the application lab spikes was 97 percent (low) and 102 percent (high).
- 2) <u>Trip Spikes:</u> The trip spike results for the application study are listed in Table 5 of Appendix II (page 84 of Appendices). Four canisters were spiked with carbon disulfide, two at 7.65 ug/m³ (low spike level) and 38.1 ug/m³ (high spike level). The average recovery for carbon disulfide for the application trip spikes was 99 percent (low) and 104% (high). These results are consistent with the lab spike results and indicate that the sample transport, storage and analytical procedures used in this study produce acceptable results for carbon disulfide.
- 3) <u>Field Spikes:</u> The field spike results for the application study are listed in Table 4 of Appendix II (page 84 of Appendices). Four canisters were spiked with carbon disulfide, two at 7.65 ug/m³ (low spike level) and 38.1 ug/m³ (high spike level). The average recovery for carbon disulfide for the application field spikes was 99% (low) and 102% (high). The field spike results are consistent with the lab and trip spike results and indicate that the sampling, sample transport, storage and analytical procedures used in this study produce acceptable results for carbon disulfide.

Figure 1, Cartridge and Canister Sampling Manifolds



Figure 2

Metam Sodium Application Site
Ventura County

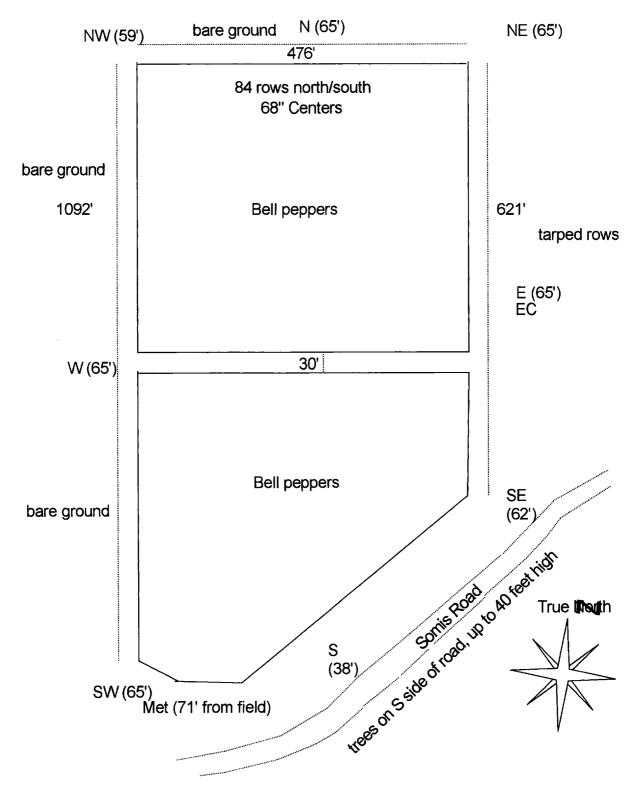


Figure 3, Application Site Area Map

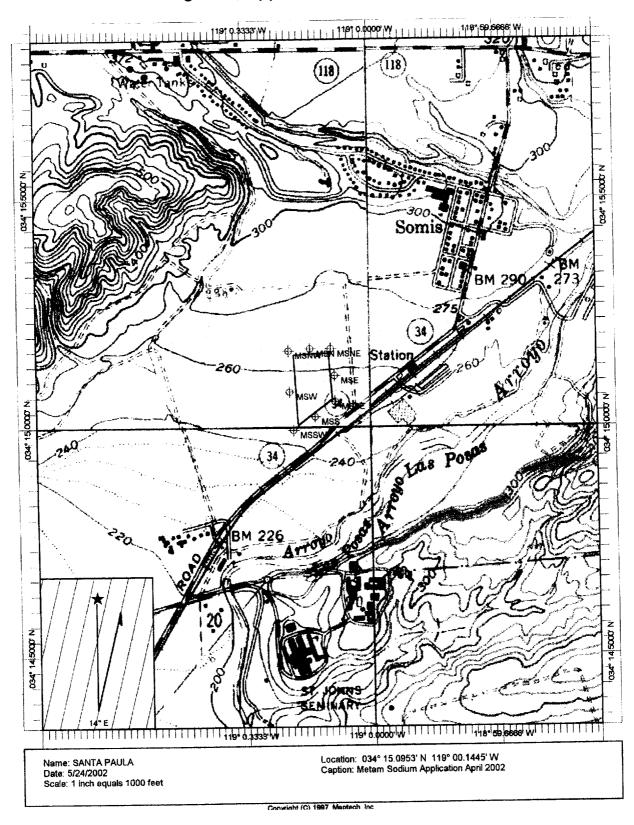
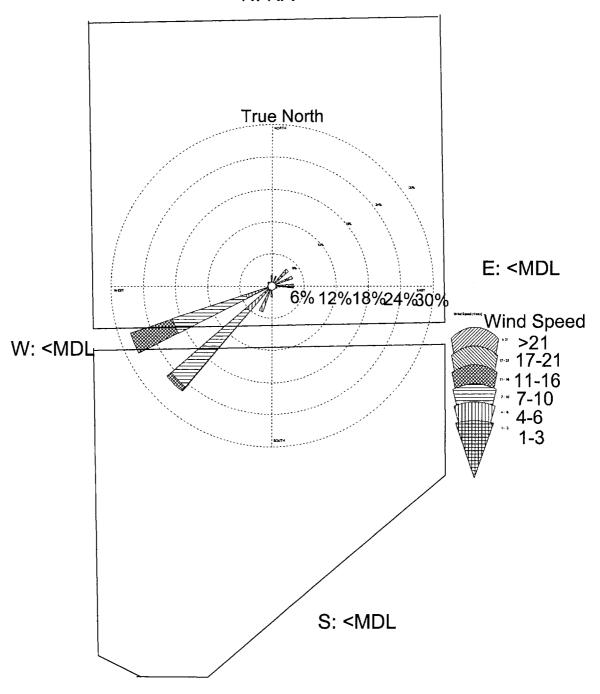


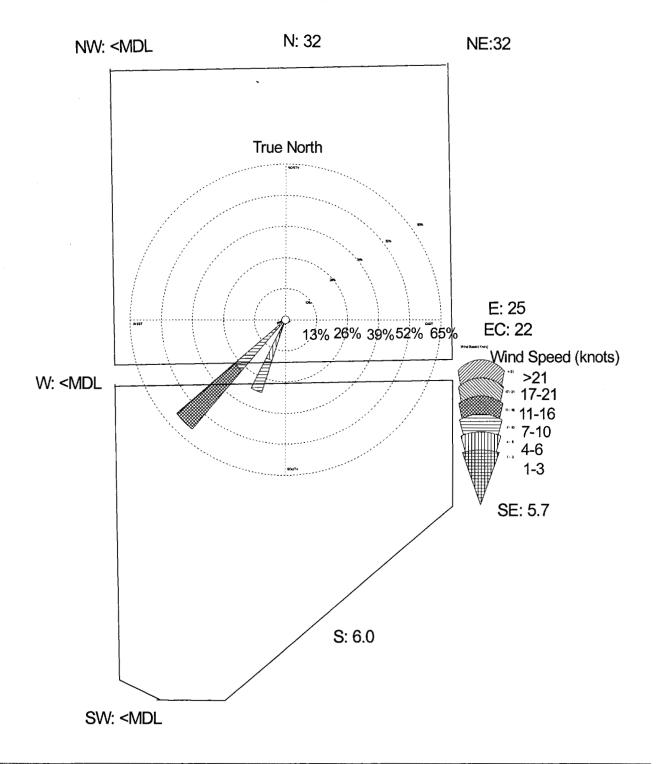
Figure 4
MITC Air Monitoring Results (ug/m³)
Background Period

N: NA



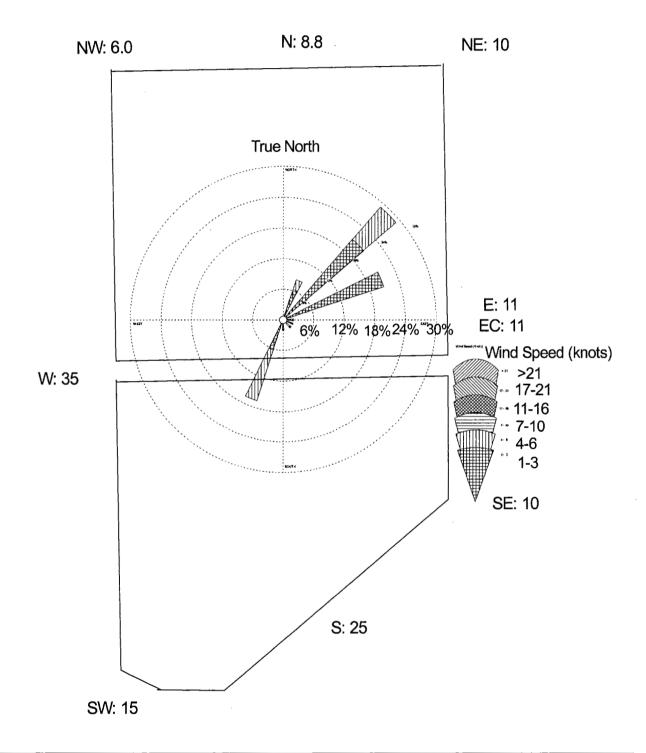
Company Name	• •		Sample Date-Time		
ARB	ARB Direction (blowing from)		5/7/02 1000 to 5/8/02 1015		
Display	Units	Calm Winds	Sample ID		
Wind Speed	Knots	21%	Background Period		

Figure 5
MITC Air Monitoring Results (ug/m³)
Period 2



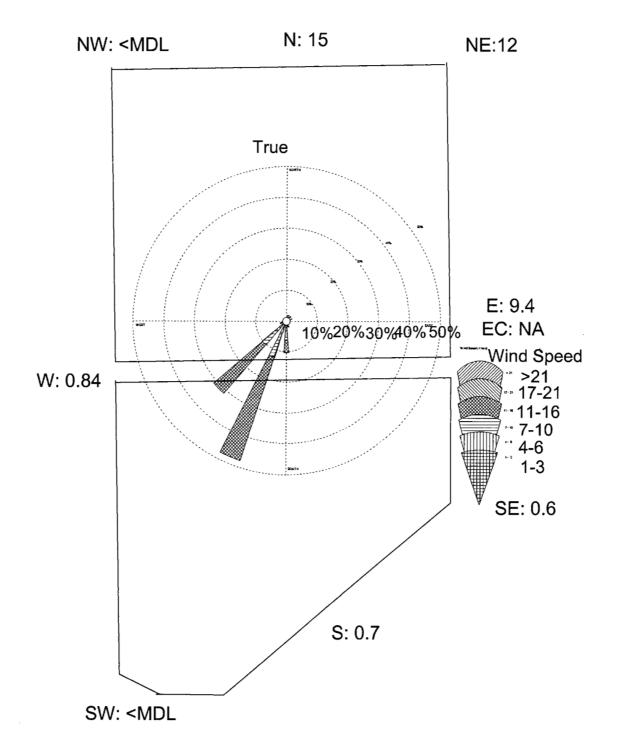
Company Name	Orientation	Avg. Wind Speed	Sample Date-Time
ARB	Direction (blowing from)	8.72 Knots	5/9/02 0700 to 5/9/02 1900
Display	Units	Calm Winds	Sample ID
Wind Speed	Knots	0%	Period 2

Figure 6
MITC Air Monitoring Results (ug/m³)
Period 3



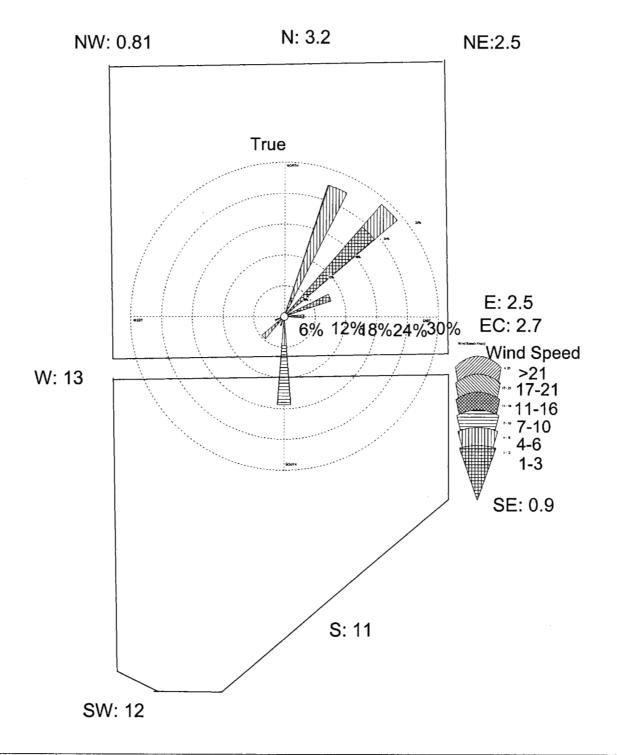
Company Name	Orientation	Avg. Wind Speed	Sample Date-Time
ARB	Direction (blowing from)	2.79 Knots	5/9/02 1900 to 5/10/02 0630
Display	Units	Calm Winds	Sample ID
Wind Speed	Knots	17%	Period 3

Figure 7
MITC Air Monitoring Results (ug/m³)
Period 4



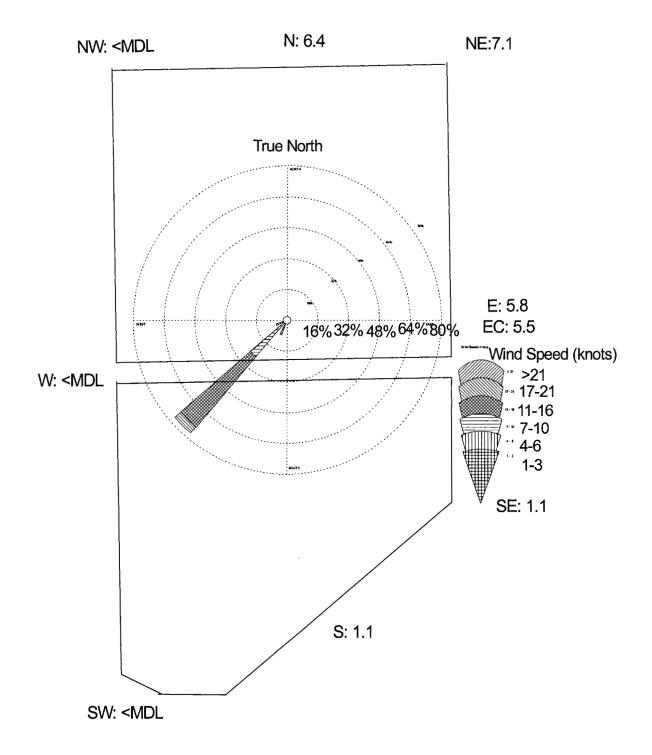
Company Name	Company Name Orientation ARB Direction (blowing from)		Sample Date-Time 5/10/02 0630 to 5/10/02 1800
Display	Units	10.28 Knots Calm Winds	Sample ID
Wind Speed	Knots	0%	Period 4

Figure 8
MITC Air Monitoring Results (ug/m³)
Period 5



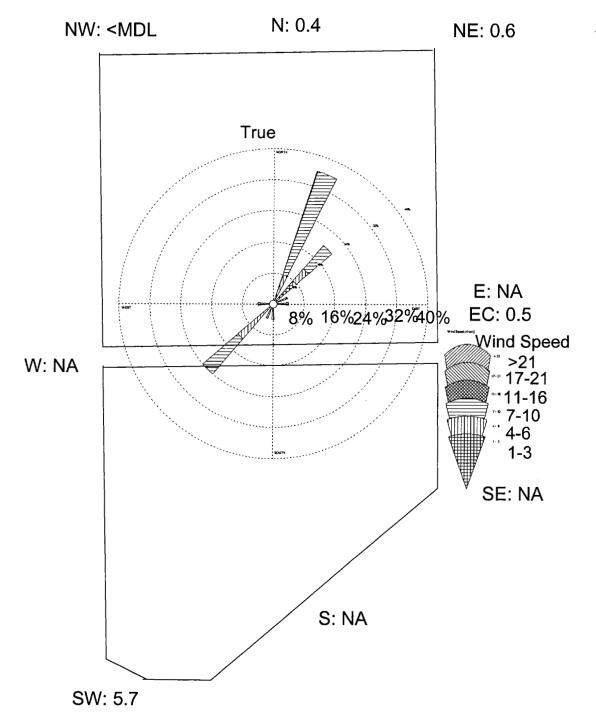
Company Name	Orientation	Avg. Wind Speed	Sample Date-Time
ARB	Direction (blowing from)	3.97 Knots	5/10/02 1800 to 5/11/02 0630
Display	Units	Calm Winds	Sample ID
Wind Speed	Knots	4%	Period 5

Figure 9
MITC Air Monitoring Results (ug/m³)
Period 6



		Avg. Wind Speed	Sample Date-Time
ARB	Direction (blowing from)	2.08 Knots	5/11/02 0630 to 5/11/02 1800
Display			Sample ID
Wind Speed	Knots	11%	Period 6

Figure 10
MITC Air Monitoring Results (ug/m³)
Period 7



Company Name ARB	Orientation Direction (blowing from)	Avg. Wind Speed 5.80 Knots	Sample Date-Time 5/11/02 1800 to 5/12/02 0630
Display	Units	Calm Winds	Sample ID
Wind Speed	Knots	0%	Period 7

Table 5. MITC Monitoring Results From Metam Sodium Application

										_
Log #	Sample ID	Start Date/Time	End Date/Time	Time (min)	Time (hours)	Volume (m3)	MITC (ug/sample)	(ug/m3)	**(ppbv)	
2	MIWB1	05/07/02 0943	05/08/02 0943	1440	24.0	3.41	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<>	<mdl< td=""><td>1</td></mdl<>	1
4	MISB1	05/07/02 1000	05/08/02 1018	1458	24.3	3.65	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<>	<mdl< td=""><td>1</td></mdl<>	1
6	MIEB1	05/07/02 1013	05/08/02 1034	1461	24.4	3.65	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<>	<mdl< td=""><td>1</td></mdl<>	1
8	MINB1	05/07/02 1025	05/08/02 1054	1469	24.5	NA	NA	NA		12
9	MIW2	05/09/02 0703	05/09/02 1810	667	11.1	1.67	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<>	<mdl< td=""><td>1</td></mdl<>	1
10	MIS2	05/09/02 0708	05/09/02 1830	682	11.4	1.71	10.2	6.0E+00	2.0E+00	1
11	MIEC2	05/09/02 0712	05/09/02 1905	713	11.9	1.78	39.0	2.2E+01	7.3E+00	1
	MIE2	05/09/02 0712	05/09/02 1900	708	11.8	1.77	44.4	2.5E+01	8.4E+00	1
	MIN2	05/09/02 0718	05/09/02 1935	737	12.3	1.84	59.3	3.2E+01	1.1E+01	İ
14	MINW2	05/09/02 0700	05/09/02 1800	660	11.0	1.65	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<>	<mdl< td=""><td>1</td></mdl<>	1
	MINE2	05/09/02 0715	05/09/02 1923	728	12.1	1.82	58.4	3.2E+01	1.1E+01	1
	MISE2	05/09/02 0710	05/09/02 1850	700	11.7	1.75	9.90	5.7E+00	1.9E+00	1
	MISW2	05/09/02 0705	05/09/02 1820	675	11.3	1.59	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<>	<mdl< td=""><td>1</td></mdl<>	1
18	MINW3	05/09/02 1807	05/10/02 0548	701	11.7	1.75	10.6	6.0E+00	2.0E+00	1
	MIW3	05/09/02 1818	05/10/02 0557	699	11.7	1.75	60.7	3.5E+01	1.2E+01	1
	MISW3	05/09/02 1825	05/10/02 0610	705	11.8	1.76	26.7	1.5E+01	5.1E+00	ĺ
	MIS3	05/09/02 1848	05/10/02 0615	687	11.4	1.72	43.1	2.5E+01	8.4E+00	1
	MISE3	05/09/02 1857	05/10/02 0623	686	11.4	1.72	17.6	1.0E+01	3.4E+00	1
	MIE3	05/09/02 1917	05/10/02 0630	673	11.2	1.68	19.1	1.1E+01	3.8E+00	1
	MIEC3	05/09/02 1917	05/10/02 0630	673	11.2	1.68	18.2	1.1E+01	3.6E+00	1
	MINE3	05/09/02 1930	05/10/02 0645	675	11.3	1.69	17.6	1.0E+01	3.5E+00	İ
	MIN3	05/09/02 1943	05/10/02 0653	670	11.2	1.68	14.7	8.8E+00	2.9E+00	
	MINW4	05/10/02 0553	05/10/02 1715	682	11.4	1.71	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td>l</td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td>l</td></mdl<></td></mdl<>	<mdl< td=""><td>l</td></mdl<>	l
	MIW4	05/10/02 0600	05/10/02 1725	685	11.4	1.71	1.44	8.4E-01	2.8E-01	
	MISW4	05/10/02 0613	05/10/02 1735	682	11.4	1.71	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td></td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td></td></mdl<></td></mdl<>	<mdl< td=""><td></td></mdl<>	
	MIS4	05/10/02 0620	05/10/02 1745	685	11.4	1.71	1.2	7.0E-01	2.3E-01	
31	MISE4	05/10/02 0625	05/10/02 1757	692	11.5	1.73	1.1	6.4E-01	2.1E-01	

^{*}Data flag; see Page 26 for details

^{**}ppbv at 1 atm and 25 C

MDL = 0.1ug/sample

Det = Value was below the EQL of 0.5 ug/sample but ≥MDL NA = Not Applicable (sampling problem)

Table 5. MITC Monitoring Results From Metam Sodium Application

Log #	Sample ID	Start Date/Time	End Date/Time	Time (min)	Time (hours)	Volume (m3)	MITC (ug/sample)	(ug/m3)	**(ppbv)	
32	MIE4	05/10/02 0635	05/10/02 1808	693	11.6	1.73	16.3	9.4E+00	3.1E+00	}
33	MIEC4	05/10/02 0640	05/10/02 1815	695	11.6	NA	NA	NA	NA	2
34	MINE4	05/10/02 0648	05/10/02 1830	702	11.7	1.76	21.7	1.2E+01	4.1E+00]
35	MIN4	05/10/02 0655	05/10/02 1840	705	11.8	1.76	25.6	1.5E+01	4.8E+00]
36	MINW5	05/10/02 1720	05/11/02 0544	744	12.4	1.86	1.50	8.1E-01	2.7E-01]
37	MIW5	05/10/02 1730	05/11/02 0552	742	12.4	1.85	23.5	1.3E+01	4.2E+00]
38	MISW5	05/10/02 1740	05/11/02 0603	743	12.4	1.86		1.2E+01	4.0E+00]
39	MIS5	05/10/02 1752	05/11/02 0610	738	12.3	1.85	21.2	1.1E+01	3.8E+00]
40	MISE5_	05/10/02 1802	05/11/02 0622	740	12.3	1.74	1.56	9.0E-01	3.0E-01]1
41	MIE5	05/10/02 1815	05/11/02 0632	_737	12.3	1.84	4.59	2.5E+00	8.3E-01]
42	MIEC5	05/10/02 1822	05/11/02 0640	738	12.3	1.85	4.95	2.7E+00	9.0E-01	1
43	MINE5	05/10/02 1835	05/11/02 0650	735	12.2	1.84	4.68	2.5E+00	8.5E-01	1
44	MIN5	05/10/02 1848	05/11/02 0659	731	12.2	1.83	5.85	3.2E+00	1.1E+00	1
45	MINW6	05/11/02 0547	05/11/02 1855	788	13.1	NA	NA	NA	NA	2
46	MIW6	05/11/02 0558	05/11/02 1702	664	11.1	1.66	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<>	<mdl< td=""><td>1</td></mdl<>	1
47	MISW6	05/11/02 0607	05/11/02 1727	680	11.3	1.70	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<>	<mdl< td=""><td>1</td></mdl<>	1
48	MIS6	05/11/02 0618	05/11/02 1737	679	11.3	1.70	1.86	1.1E+00	3.7E-01	1
49	MISE6	05/11/02 0626	05/11/02 1750	684	11.4	1.71	1.92	1.1E+00	3.8E-01	1
50	MIE6	05/11/02 0638	05/11/02 1806	688	11.5	1.72	9.90	5.8E+00	1.9E+00	1
51	MIEC6	05/11/02 0645	05/11/02 1816	691	11.5	1.56	8.52	5.5E+00	1.8E+00	1
52	MINE6	05/11/02 0655	05/11/02 1834	699	11.6	1.75	12.4	7.1E+00	2.4E+00	1
53	MIN6	05/11/02 0705	05/11/02 1841	696	11.6	1.74	11.1	6.4E+00	2.1E+00	1
54	MINW7	05/11/02 1900	05/12/02 0526	626	10.4	1.57	<mdl< td=""><td><mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<></td></mdl<>	<mdl< td=""><td><mdl< td=""><td>1</td></mdl<></td></mdl<>	<mdl< td=""><td>1</td></mdl<>	1
55	MIW7	05/11/02 1707	05/12/02 0535	748	12.5	NA	NA	NA	NA	2
56	MISW7	05/11/02 1730	05/12/02 0551	741	12.4	1.74	10.6	6.1E+00	2.0E+00	1
57	MIS7	05/11/02 1742	05/12/02 0603	741	12.3	NA	NA	NA	NA	2

^{*}Data flag; see Page 26 for details

^{**}ppbv at 1 atm and 25 C

MDL = 0.1ug/sample

Det = Value was below the EQL of 0.5 ug/sample but ≥MDL

NA = Not Applicable (sampling problem)

Table 5. MITC Monitoring Results From Metam Sodium Application

Log #	Sample ID	Start Date/Time	End Date/Time	Time (min)	Time (hours)	Volume (m3)	MITC (ug/sample)	(ug/m3)	**(ppbv)	
58	MISE7	05/11/02 1756	05/12/02 0611	735	12.3	NA	NA	NA	NA	2
59	MIE7	05/11/02 1812	05/12/02 0626	734	12.2	NA	NA	NA	NA	2
60	MIEC7	05/11/02 1823	05/12/02 0627	724	12.1	1.68	0.9	5E-01	2E-01]1
61	MINE7	05/11/02 1839	05/12/02 0650	731	12.2	1.83	1.1	6.0E-01	2.0E-01]
62	MIN7	05/11/02 1845	05/12/02 0656	731	12.2	1.83	0.7	4E-01	1E-01]

Data Flags: 1. End flow rate was >10% but <25% from 2.5 slpm; start and end flow rates averaged. 2. Invalid sample due to field sampling problem; end flow rate > 25% from 2.5 slpm.

MDL = 0.1ug/sample

Det = Value was below the EQL of 0.5 ug/sample but ≥MDL

NA = Not Applicable (sampling problem)

^{*}Data flag; see Page 26 for details

^{**}ppbv at 1 atm and 25 C

Table 6. Summary of MITC Monitoring Results From Metam Sodium Application (ug/m3)

Sampling Period	MIE (east)	MIEC (east collocated)	MIN (north)	MINE (N-east)	MINW (N-west)	MIS (south)	MISE (S-east)	MISW (S-west)	MIW (west)
Background	<mdl< td=""><td></td><td>NA</td><td></td><td></td><td><mdl< td=""><td></td><td></td><td><mdl< td=""></mdl<></td></mdl<></td></mdl<>		NA			<mdl< td=""><td></td><td></td><td><mdl< td=""></mdl<></td></mdl<>			<mdl< td=""></mdl<>
Period 2	2.5E+01	2.2E+01	3.2E+01	3.2E+01	<mdl< td=""><td>6.0E+00</td><td>5.7E+00</td><td><mdl< td=""><td><mdl< td=""></mdl<></td></mdl<></td></mdl<>	6.0E+00	5.7E+00	<mdl< td=""><td><mdl< td=""></mdl<></td></mdl<>	<mdl< td=""></mdl<>
Period 3	1.1E+01	1.1E+01	8.8E+00	1.0E+01	6.0E+00	2.5E+01	1.0E+01	1.5E+01	3.5E+01
Period 4	9.4E+00	NA	1.5E+01	1.2E+01	<mdl< td=""><td>7.0E-01</td><td>6.4E-01</td><td><mdl< td=""><td>8.4E-01</td></mdl<></td></mdl<>	7.0E-01	6.4E-01	<mdl< td=""><td>8.4E-01</td></mdl<>	8.4E-01
Period 5	2.5E+00	2.7E+00	3.2E+00	2.5E+00	8.1E-01	1.1E+01	9.0E-01	1.2E+01	1.3E+01
Period 6	5.8E+00	5.5E+00	6.4E+00	7.1E+00	NA	1.1E+00	1.1E+00	<mdl< td=""><td><mdl< td=""></mdl<></td></mdl<>	<mdl< td=""></mdl<>
Period 7	NA	5E-01	4E-01	6.0E-01	<mdl< td=""><td>NA</td><td>NA</td><td>5.7E+00</td><td>NA</td></mdl<>	NA	NA	5.7E+00	NA

Table 7. MITC Collocated Monitoring Results (ug/m3)

Sampling Period	MIE (east)	MIEC (east collocated)	Average	Relative Percent Difference
Period 2	2.5E+01	2.2E+01	2.3E+01	14
Period 3	1.1E+01	1.1E+01	1.1E+01	5
Period 4	9.4E+00	NA	NA	NA
Period 5	2.5E+00	2.7E+00	2.6E+00	7
Period 6	5.8E+00	5.5E+00	5.6E+00	5
Period 7	NA	5E-01	NA	NA

MDL = 0.1 ug/sample

Det = Value was below the EQL of 0.5 ug/sample but ≥MDL NA = Not Applicable (sampling problem)